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TITLE: MEASUREMENT OF SELECTED CHEMICALS IN SOIL FROM THE  
DEAD CREEK SITE - ILLINOIS EPA SPLIT SAMPLES

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ABSTRACT: Three sediment samples and one well water sample were taken on October 2, 1980 by Monsanto and IEPA representatives. The Monsanto samples were transferred to our laboratory and analyzed for polychlorinated biphenyls, elemental phosphorus, chlorobenzenes, chlorophenols, phosphate esters, and metals (including arsenic and inorganic phosphorus). No elemental phosphorus was detected in any of the samples, which implies that phosphorus is not responsible for the "smoking earth" reported at the site. In addition, no organic chemicals were detected above the detection limits in the well water sample. However, varying amounts of the organic chemicals and metals were measured in the soil samples. One sample contained higher levels of polychlorinated biphenyls and other organic compounds, while the other two samples contained higher levels of metals. The results clearly indicate non-uniform contamination at the Dead Creek site.

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REPT. NO.: ES-80-SS-24  
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R-1080(A)

MEASUREMENT OF SELECTED CHEMICALS IN SOIL FROM THE DEAD CREEK SITE  
ILLINOIS EPA SPLIT SAMPLESINTRODUCTION

Following media reports and subsequent Illinois EPA concern about hazardous chemicals at the Dead Creek site near Sauget, Illinois, personnel from Monsanto's W. G. Krummrich Plant and the Illinois EPA sampled several areas at the site and split the samples. The Monsanto samples were submitted to Environmental Sciences for characterization. Monsanto's concerns about the site arose from reports of high levels of polychlorinated biphenyls and phosphorus, as well as the reported presence of other chemicals, and the proximity of the site to the Krummrich Plant. These samples were taken to give both Monsanto and the Illinois EPA opportunity to confirm the reported levels found in earlier samplings by the Illinois EPA. In addition to polychlorinated biphenyls and phosphorus, several other "families" of chemicals were measured to try to identify or eliminate possible sources of the chemicals at the site.

SUMMARY

Three sediment samples and one well water sample were taken on October 2, 1980 by Monsanto and IEPA representatives. The Monsanto samples were transferred to our laboratory and analyzed for polychlorinated biphenyls, elemental phosphorus, chlorobenzenes, chlorophenols, phosphate esters, and metals (including arsenic and inorganic phosphorus). No elemental phosphorus was detected in any of the samples, which implies that phosphorus is not responsible for the "smoking earth" reported at the site. In addition, no organic chemicals were detected above the detection limits in the well water sample. However, varying amounts of the organic chemicals and metals were measured in the soil samples. One sample contained higher levels of polychlorinated biphenyls and other organic compounds, while the other two samples contained higher levels of metals. The results clearly indicate non-uniform contamination at the Dead Creek site.

DETAILSSampling

The three soil and one water samples were collected by Monsanto W. G. Krummrich plant personnel and IEPA personnel and split at the site. The Monsanto samples were transferred to the Environmental Analysis Group. In our laboratory, the sediment samples were handled according to Standard Operating Procedure (SOP) EAN-80-SOP-6, Homogenizing, Subdividing and Preserving Sediment Samples. Portions of the soil samples were transferred to Applied Sciences for the determination of metals and arsenic.

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### Analytical Procedures

The three soil samples were analyzed for a variety of chemicals using established procedures or methods developed and validated for the chemicals of interest in soil. The following list tabulates the methods which were used.

| Analyte                                | Method No. | Title  |
|--|------------|--|
| Polychlorinated Biphenyls              | ES-80-M-28 | Determination of Polychlorinated Biphenyls in Soil and Sediment                              |
| Chlorinated Benzenes                   | ES-80-M-29 | Determination of Chlorinated Benzenes in Soil and Sediment                                   |
| Chlorinated Phenols                    | ES-80-M-30 | Determination of Chlorinated Phenols in Soil and Sediment                                    |
| Elemental Phosphorus (P <sub>4</sub> ) | ES-80-M-24 | Determination of Elemental Phosphorus (P <sub>4</sub> ) in Soil and Sediment                 |
| Phosphate Esters                       | ES-80-M-5  | Determination of Group I Compounds in Sediments . . .  |
| Metals                                 | Ref. 1, 2  | Inductively Coupled Plasma (ICP) . . . Method for Trace Element Analysis of Water and Wastes |
| Arsenic                                | Ref. 3     | Methods for Chemical Analysis of Water and Wastes-Arsenic                                    |

All determinations were carried out in strict accordance with these methods, except that the polychlorinated biphenyls, chlorinated benzenes and phosphate esters were measured in extracts from acidified samples to facilitate determination of chlorinated phenols in the same extracts.

The water sample was extracted in accordance with SOP EAN-80-SOP-19, Extraction of Semivolatile Organic Compounds from Water. The levels of polychlorinated biphenyls and phosphorus were determined using the analytical conditions specified in the respective method for soils listed above.

### Results

The analytical results for this study are tabulated in Tables I-VI. Each table contains the results for all of the samples for a specific group of compounds. All results for the soils are in ppm (parts per million or ug/g). The results for the water sample are in ppb (parts per billion, ng/g). In general, the stated detection limits are the lowest level at which a given measurement was validated. Levels which are apparently real, but which are below the validated detection limit are presented in parentheses.

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Quality Assurance

The quality assurance results (i.e., recovery and precision evaluations) for these samples have been compiled along with those of similar samples analyzed concurrently. These results are reported in Special Study ES-80-SS-27, Measurement of Selected Chemicals in Soil from the Dead Creek Site - Quality Assurance.

REFERENCES

1. Methods for Chemical Analysis of Waters and Wastes, EPA-600/4-79-020, page: Metals-6, Section 4.1.3.
2. Federal Register, Vol. 44, No. 233, December 3, 1979.
3. Methods for Chemical Analysis of Waters and Wastes, EPA-600/4-79-020, Method 206-Arsenic, pages: 206.2-1 to 206.5-2.

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TABLE 1. PPM LEVELS OF PCBs AND ELEMENTAL PHOSPHORUS (P<sub>4</sub>) IN DEAD CREEK SOIL AND WATER SAMPLES

| ANALYTE | ES LOG NO.<br>DATE SAMPLED<br>LOCATION | 0100301<br>10/2/80<br>40 yds south<br>of Queeny Ave.<br>Center of Creek |  | 0100303<br>10/2/80<br>268 paces<br>south of<br>0100301 |  | 0100305<br>10/2/80<br>270 paces<br>south of<br>0100303 |  | (Water)<br>0100307<br>10/2/80<br>Well at Theresa's<br>Greenhouse, 101<br>Walnut, Sauget, IL. |  | 0041701<br>4/16/80<br>Soil Blank<br>Mo. Bottoms<br>St. Charles, MO. |  |
|---------|--|---|--|--|--|--|--|--|--|---|--|
|         |  | 0100301<br>10/2/80<br>40 yds south<br>of Queeny Ave.<br>Center of Creek |  | 0100303<br>10/2/80<br>268 paces<br>south of<br>0100301 |  | 0100305<br>10/2/80<br>270 paces<br>south of<br>0100303 |  | (Water)<br>0100307<br>10/2/80<br>Well at Theresa's<br>Greenhouse, 101<br>Walnut, Sauget, IL. |  | 0041701<br>4/16/80<br>Soil Blank<br>Mo. Bottoms<br>St. Charles, MO. |  |

|  |   |         |         |        |            |        |
|--|---|---------|---------|--------|------------|--------|
| PCB's (Cl <sub>2</sub> to<br>Cl <sub>6</sub> Homologs) | IEPA (public release)                       | 13,000  | 240     | 45     | ND < 1 ppb | ND < 1 |
|  | IEPA (public release)                       | 10,000  | 360     | 73     |            |        |
|  | IEPA (public release)<br>but per conference | 17,000  | 130     | 59     |            |        |
| P <sub>4</sub>   | IEPA (public release)                       | ND < 1  | ND < 1  | ND < 1 | ND < 1 ppb | ND < 1 |
|  |   | 130,000 | 27,000  | 2000   |            |        |
|  |   | 5000    | 160,000 | 93,000 |            |        |
| Total Phos   |   | 10,000  | 350     | 73     | < 1 ppb    |        |
| (Min.)   |   | 2000    | 8900    | 4700   | < 1 ppb    |        |

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TABLE II. PPM LEVELS OF CHLOROBENZENES IN DEAD CREEK SOIL SAMPLES

| ANALYTE                      | ES LOG NO.<br>DATE SAMPLED<br>LOCATION | 0100301<br>10/2/80<br>40 yds south<br>of Queeny Ave.<br>Center of Creek | 0100303<br>10/2/80<br>268 paces<br>south of<br>0100301 | 0100305<br>10/2/80<br>270 paces<br>south of<br>0100303 | 0041701<br>4/16/80<br>Soil Blank<br>Mo. Bottoms<br>St. Charles, MO. |
|------------------------------|--|---|--|--|---|
| MONOCHLOROBENZENE            |  | (0.9)   | ND < 1   | (0.3)  | ND < 1  |
| P-DICHLOROBENZENE            |  | 370   | (0.3)  | (0.4)  | ND < 1  |
| O-DICHLOROBENZENE            |  | 80  | (0.6)  | 1.0  | ND < 1  |
| TRICHLOROBENZENES (3)        |  | 85  | 1.6  | (0.7)  | ND < 1  |
| TETRACHLOROBENZENES (3)      |  | 6.1   | 2.4  | (0.4)  | ND < 1  |
| PENTACHLOROBENZENE           |  | ND < 1  | ND < 1   | ND < 1   | ND < 1  |
| HEXACHLOROBENZENE            |  | ND < 1  | 1.2  | ND < 1   | ND < 1  |
| NITROCHLOROBENZENES (O-, P-) |  | 120   | ND < 1   | ND < 1   | ND < 1  |

( ) Values in parentheses are below the validated detection limit. However, they represent levels detected with a S/N > 2.5 and can be considered semi-quantitative.

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TABLE III. PPM LEVELS OF CHLOROPHENOLS IN DEAD CREEK SOIL SAMPLES

| ANALYTE            | ES LOG NO.<br>DATE SAMPLED<br>LOCATION | 0100301<br>10/2/80<br>40 yds south<br>of Queeny Ave.<br>Center of Creek | 0100303<br>10/2/80<br>268 paces<br>south of<br>0100301 | 0100305<br>10/2/80<br>270 paces<br>south of<br>0100303 | 0041701<br>4/16/80<br>Soil Blank<br>Mo. Bottoms<br>St. Charles, MO. |
|--------------------|--|---|--|--|---|
|                    |  |   |  |  |   |
| O-CHLOROPHENOL     |  | 3.7   | ND < 1   | ND < 1   | ND < 1  |
| P-CHLOROPHENOL     |  | 6.6   | ND < 1   | (0.9)  | ND < 1  |
| 2,4-DICHLOROPHENOL |  | 1.2   | ND < 1   | ND < 1   | ND < 1  |
| PENTACHLOROPHENOL  |  | 130   | ND < 1   | 1.8  | ND < 1  |

( ) Values in parentheses are below the validated detection limit. However, they represent levels detected with a S/N >2.5 and can be considered semi-quantitative.

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TABLE IV. PPM LEVELS OF PHOSPHATE ESTERS IN DEAD CREEK SOIL SAMPLES

| ANALYTE                               | ES LOG NO.<br>DATE SAMPLED<br>LOCATION | 0100301  | 0100303                                     | 0100305                                     | 0041701  |
|---------------------------------------|--|--|---|---|--|
|                                       |  | 10/2/80<br>40 yds south<br>of Queeny Ave.<br>Center of Creek | 10/2/80<br>268 paces<br>south of<br>0100301 | 10/2/80<br>270 paces<br>south of<br>0100303 | 4/16/80<br>Soil Blank<br>Mo. Bottoms<br>St. Charles, MO. |
| DIBUTYLPHENYL<br>PHOSPHATE            |  | 330  | ND < 1                                      | (0.8)                                       | ND < 1   |
| BUTYLDIPHENYL<br>PHOSPHATE            |  | ND < 1   | ND < 1                                      | (0.8)                                       | ND < 1   |
| TRIPHENYL<br>PHOSPHATE                |  | 2600   | ND < 1                                      | ND < 1                                      | ND < 1   |
| 2-ETHYLHEXYLDIPHENYL<br>PHOSPHATE     |  | ND < 1   | ND < 1                                      | 2.2   | ND < 1   |
| ISODECYLDIPHENYL<br>PHOSPHATE         |  | ND < 1   | ND < 1                                      | ND < 1                                      | ND < 1   |
| T-BUTYLPHENYLDIPHENYL<br>PHOSPHATE    |  | 28   | ND < 1                                      | ND < 1                                      | ND < 1   |
| DI-T-BUTYLPHENYLDIPHENYL<br>PHOSPHATE |  | ND < 1   | ND < 1                                      | ND < 1                                      | ND < 1   |
| NONYLPHENYLDIPHENYL<br>PHOSPHATE      |  | ND < 1   | ND < 1                                      | ND < 1                                      | ND < 1   |
| CUMYLPHENYLDIPHENYL<br>PHOSPHATE      |  | 3.7  | ND < 1                                      | ND < 1                                      | ND < 1   |

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( ) Values in parentheses are below the validated detection limit. However, they represent levels detected with a S/N > 2.5 and can be considered semi-quantitative.

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TABLE V. PPM LEVELS OF METALS IN DEAD CREEK SOIL SAMPLES

| ANALYTE    | ES LOG NO.<br>DATE SAMPLED<br>LOCATION | 0100301<br>10/2/80<br>40 yds south<br>of Queeny Ave.<br>Center of Creek | 0100303<br>10/2/80<br>268 paces<br>south of<br>0100301 | 0100305<br>10/2/80<br>270 paces<br>south of<br>0100303 | 0041701<br>4/16/80<br>Soil Blank<br>Mo. Bottoms<br>St. Charles, MO. |
|------------|--|---|--|--|---|
| SILVER     |  | ND <1   | 42   | 29   | ND <1   |
| ALUMINUM   |  | 1400  | 5100   | 5300   | 5600  |
| BARIUM     |  | 770   | 1200   | 1300   | 130   |
| BERYLLIUM  |  | ND <1   | ND <1  | ND <1  | ND <1   |
| BORON      |  | 28  | 160  | 100  | 27  |
| CALCIUM    |  | 8500  | 9200   | 6200   | 4600  |
| CADMIUM    |  | 5.1   | 60   | 55   | 3.9   |
| COBALT     |  | 15  | 180  | 120  | 33  |
| CHROMIUM   |  | 25  | 110  | 240  | 19  |
| COPPER     |  | 460   | 28,000   | 18,000   | 19  |
| IRON       |  | 4700  | 53,000   | 30,000   | 9900  |
| MAGNESIUM  |  | 460   | 2200   | 2000   | 2300  |
| MANGANESE  |  | 29  | 170  | 110  | 510   |
| MOLYBDENUM |  | 6.1   | 92   | 68   | 11  |
| SODIUM     |  | 400   | 540  | 410  | 320   |
| NICKEL     |  | 110   | 2000   | 1700   | 39  |
| LEAD       |  | 180   | 2000   | 1600   | 50  |
| PHOSPHORUS |  | 2500  | 13,000   | 9400   | 610   |
| ANTIMONY   |  | 13  | 240  | 160  | 29  |
| SILICON    |  | 73  | 150  | 89   | 110   |
| TIN        |  | 18  | 260  | 220  | 18  |
| STRONTIUM  |  | 35  | 230  | 110  | 17  |
| TITANIUM   |  | 32  | 110  | 80   | 37  |
| VANADIUM   |  | 34  | 140  | 130  | 130   |
| ZINC       |  | 280   | 32,000   | 18,000   | 56  |

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TABLE VI. SUMMARY OF PHOSPHORUS CONTENT (PPM) OF DEAD CREEK SOIL SAMPLES

| ANALYTE                             | ES LOG NO.<br>DATE SAMPLED<br>LOCATION | 0100301<br>10/2/80<br>40 yds south<br>of Queeny Ave.<br>Center of Creek | 0100303<br>10/2/80<br>268 paces<br>south of<br>0100301 | 0100305<br>10/2/80<br>270 paces<br>south of<br>0100305 | 0041701<br>4/16/80<br>Soil Blank<br>Mo. Bottoms<br>St. Charles, MO. |
|-------------------------------------|--|---|--|--|---|
|                                     |  |   |  |  |   |
| P - ELEMENTAL,<br>By GC/MS          |  | ND < 1  | ND < 1   | ND < 1   | ND < 1  |
| P-INORGANIC,<br>By ICP              |  | 2500  | 13,000   | 9400   | 610   |
| TOTAL PHOSPHATE<br>ESTERS, By GC/MS |  | 3000  | ND < 10  | 4  | ND < 10   |
| Phosphorus (releant)                |  | 25,000  | 160,000  | 93,000   |   |
| Phosphorus (just testing)<br>by EPA |  | 2,000   | 120,000  | 32,100   |   |

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